

**METHOD AND APPARATUS FOR THE DRYING OF
CEMENTED CARBIDE POWDER AND THE LIKE**

This application is a divisional application of Application No. 09/787,718,
filed on May 31, 2001, which was a national stage filing under 35 U.S.C. §371 of
5 International Application No. PCT/SE99/01698 filed on September 24, 1999, and
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FIELD OF THE INVENTION

[01] The present invention relates to cemented carbides and a method and
device for drying powder mixtures of cemented carbide and the like.

10 **BACKGROUND OF THE INVENTION**

[02] In the discussion of the state of the art that follows, reference is made to
certain structures and/or methods. However, the following references should not
be construed as an admission that these structures and/or methods constitute prior
art. Applicant expressly reserves the right to demonstrate that such structures
15 and/or methods do not qualify as prior art against the present invention.

[03] Cemented carbide is made by powder metallurgical methods consisting of
the wet milling in an alcohol-water-solution of a powder mixture containing
powder forming the hard constituents and a binder phase, drying of the milled
mixture to a powder with good flow properties by spray drying, pressing of the
20 dried powder to bodies of desired shape and finally sintering.

[04] The intensive milling operation is performed in mills of different sizes using cemented carbide milling bodies. Milling is considered necessary to obtain a uniform distribution of the binder phase in the milled mixture. The milling time can vary from several hours up to days. The milling operation produces a slurry
5 suitable for spray drying. Successful spray drying depends strongly on the properties of the slurry. The present technology with intensive milling under long periods of time usually gives a very fine-grained powder whose sedimentation rate is sufficiently slow. As result of the spray drying a ready-to-press powder is obtained consisting essentially of spherical agglomerates of about 0.1 mm mean
10 size.

[05] It is desirable that the spray-dried powder shall be in the form of spherical well-defined agglomerates with a limited variation of the sizes of the agglomerates. Thus, a more even powder density is obtained, giving improved flow properties as well as a reduction of filling variations in the subsequent pressing operation. The
15 distribution or size variations can be influenced by optimising the alcohol to water ratio and liquid to powder ratio in the slurry, the amount of pressing agent etc. Another way is to modify the nozzles in the spray drier. A variant of such a nozzle is described in US 4,978,069. This patent relates to the generation of even drops from a melt, but it can also be applied to drying of cemented carbide
20 powder. However, it has been found that this does not give the desired limited variation of the sizes of the agglomerates. It has been found better to use a simpler nozzle instead, and expose the powder to a fractioned sieving after the drying

process to eliminate the relatively coarse and fine part of the agglomerates to obtain the desired narrow distribution of sizes. The distribution after the sieving must be controlled since there is a risk of clogging of the net of the sieve, which may influence the results. The sieving also adversely affects the properties of the powder. However, the distribution of sizes of the agglomerates is normally so broad that the final distribution cannot be made as narrow as desirable for economical reasons. A way to describe distribution of the sizes of the agglomerates is through the expression d_{97}/d_{03} where d_{97} = the grain size below which 97 % of the agglomerates are found and d_{03} = the grain size below which 3% of the agglomerates are found. A d_{97}/d_{03} value = 4 has been found to be acceptable. However, it is desirable to obtain an even lower value, preferably without a subsequent sieving operation.

SUMMARY OF THE INVENTION

[06] It is an object of the invention to overcome the disadvantages of the prior art.

[07] It is an object of the present invention to provide a method for the production of cemented carbide powder with a limited distribution of grain sizes $d_{97}/d_{03} < 4$, without subsequent sieving.

[08] Surprisingly it has now been found that if the apparatus, according to U.S. Patent No. 4,978,069 is modified in the way more closely described below a

cemented carbide powder is obtained with the desired limited distribution of grain sizes.

[09] In an apparatus for drop forming by disks, according to the present invention, jets of the liquid are ejected towards a first funnel-shaped part where the liquid is accelerated in a first step after which the jets are permitted to spread somewhat, they are then ejected over to a second funnel-shaped part where the liquid is accelerated in a second step to the speed of the drop forming disk. The acceleration of the liquid is thus divided into two steps. During the second acceleration step the acceleration is increased because the spreading of the jet results in increased surface contact.

[010] At the periphery of the drop forming disk the risk that contact between the surface of the disk and the liquid be broken is minimized thanks to the fact that the outermost part of the disk is angled relative to the plane of rotation in such a way that the centrifugal force acting on the liquid is split into one component directed along the surface of the disk and one directed against the surface of the disk. In this way the contact between the surface of the disk and the layer of liquid right up to the means of drop forming is ensured.

[011] According to one aspect, the present invention provides a method of making powders from a slurry, the method comprising the steps of: (i) introducing the slurry into a drop forming apparatus; (ii) ejecting the slurry from a discharge opening to form a jet such that the jet hits an inwardly and downwardly oriented first oblique rotating surface; (iii) rotating the first surface at a speed

such that the jet is forced upwards over the first surface and over another connected horizontal surface by centrifugal forces; (iv) dividing and directing the jet towards an outwardly and downwardly oriented second oblique rotating surface; (v) rotating the second surface such that the slurry in the jet is accelerated to the speed of rotation of the second surface; (vi) directing the jet onto an outwardly oriented third oblique rotating surface, from which drops of the slurry detach themselves when centrifugal forces on the third surface exceeds adhesive forces between the drops and the third surface.

[012] According to another aspect, the present invention provides an apparatus for forming drops from a slurry, the apparatus comprising: a plurality of rotatable disk units, the disk units comprising a number of disks arranged axially on top on each other, the disks have a cross-section comprising a radially inner section with a substantially U-shaped recess constructed to receive a raised L-shaped section of an adjacent disk with sufficient play therebetween to allow radial ejection of a jet of the slurry; and a distributor for uniformly distributing the slurry onto the plurality of disk units.

[013] According to yet another aspect, the present invention provides powdered agglomerates of cemented carbide, cermets, ceramics or similar materials with abrasive wear resistance wherein the agglomerates have a distribution of size, prior to sieving, such that a ratio of $d_{97}/d_{03} < 4$ wherein

d_{97} = the grain size below which 97 % of the agglomerates is found and

d_{03} = the grain size below which 3 % of the agglomerates is found.

BRIEF DESCRIPTION OF THE DRAWINGS

[014] Fig 1 shows the drop forming means in a longitudinal section view;

[015] Fig 2 shows a section along the line A-A in Fig 1;

[016] Fig 3 shows part of a drop-forming disk in greater detail;

5 [017] Fig 4 shows agglomerates dried according to known technique in a 30X magnification; and

[018] Fig 5 shows agglomerates dried according to the invention in a 30X magnification.

DETAILED DESCRIPTION OF THE INVENTION

10 [019] More particularly the means of drop forming according to the invention consists of a bearing housing 1 in which a tube shaped shaft 2 is mounted, in which a shaft 3 is mounted. A belt pulley 4 and a slinger rotor 5 are mounted on the shaft 2, and a belt pulley and a lower disk 7 are mounted on the shaft 3. A distributor disk 8, to which is connected an upper disk by means of rods 13 is
15 mounted on the shaft 2. The bearing housing 1 has a flange 14 for the mounting of the drop forming apparatus in, e.g.-a drying chamber, and an in-feed tube 15 for the liquid. The belt pulley 6, the shaft 3, lower disk 7, the slinger rotor 5, the upper disk 10 and the distributor disk 8 constitute a rigidly inter-connected rotatable unit. The slinger rotor 5 has a receiving disk 16 with a groove 17 in the
20 bottom of which are formed uniformly distributed, even sized holes 18, to which are arranged axially proceeding channels 19, which are connected at their lower

ends to a disk 20. The channels 19 have discharge openings 21 arranged at the funnel-shaped part 22 of the drop forming disks. This part transforms into an essentially plane part 23 with a sharp edge 24, directed towards a second funnel-shaped part 25, which in its lower part transforms to a surface 26, which is
5 directed at an angle 27 towards the plane of rotation of the drop forming disk. The surface 26 extends radially to a sharp edge 28, on which (not shown) are arranged means of drop forming in the shape of uniformly distributed, uniform, protruding bumps.

[020] An arrangement according to one embodiment of the invention has suitably
10 the following dimensions:

[021] Radius of the distributor disk 8: 12.5 - 20 cm.

[022] Radius of the lower disk 7: 10-15 cm.

[023] Number of drop forming disks 5: 10-25.

[024] Angle of the funnel-shaped part 22: 45-55°, preferably about 50° to the
15 horizontal plane.

[025] Angle of the funnel-shaped part 25: 65-75°, preferably about 70° to the horizontal plane.

[026] Angle of the funnel-shaped part 26: < 10°, preferably about 5° to the horizontal plane.

20 [027] With these dimensions a drying capacity of 100 - 500 kg cemented carbide powder/hour is obtained.

[028] When in use, the two belt pulleys 4, 6 and thereby also the shafts 2, 3 and the means connected thereto are rotated. Liquid is supplied through the supply tube 15 and flows down onto the distributing disk 8, from which it is ejected against the groove 17. The shaft 3 is generally rotated with considerably higher speed than the shaft 2 which results in the liquid being ejected from the distributor disk 8 into the groove 17 and it is then brought to flow into the groove 17 in the direction of rotation of the distributor disk. The liquid which hits the groove 17 in a part which is limited by the farther edge of a hole 18 and that of the next hole 18 seen in the direction of the flow of the liquid and it flows out via the farther hole 18 and is led further away via the channel 19. The accuracy of the distribution is further improved in that each and every one of the holes 18 continuously occupies all the positions on the circumference. The liquid is ejected from the discharge opening 21 in the form of a jet towards the funnel-shaped part 22 of the drop-forming disk 12. The liquid is accelerated in the direction of the drop forming disk and moves in connection herewith outwards/upwards over the funnel-shaped part 22 and the plane part 23 and is ejected as a divided jet towards the other funnel shaped part 25, where the liquid is accelerated to the speed of the drop forming disk 12 and led out over the surface 26 to the means of drop forming from which the drops are released.

EXAMPLE I

[029] A cemented carbide slurry with the composition 6% Co and the balance substantially comprised of WC was dried using a drop generator according to the invention and was compared to material with corresponding composition which
5 had been spray dried with a pressure nozzle and subsequently sieved according to the known technique previously discussed. The generator according to the invention consisted of twelve disks and it had a diameter of 330 mm. The drying capacity was 450 kg/h.

[030] After drying distribution of sizes in the agglomerate was determined by
10 sieve analysis as well as laser diffraction analysis. It was found, surprisingly, that a generator according to the invention gave such a narrow distribution of sizes that a distribution factor of $d_{97}/d_{03} = 3.6$ could be obtained essentially without sieving. By contrast, powder dried with a nozzle by conventional techniques required elimination by sieving of 15 weight-% of the powder to achieve a
15 distribution factor of $d_{97}/d_{03} = 4$, and in order to achieve a distribution factor of $d_{97}/d_{03} = 3$ elimination of more than 20 weight-% of the powder by sieving is necessary.

[031] The dried powders were also studied in a microscope and it was found that powder dried according to invention exhibited not only more even agglomerate
20 size distribution, but also a better quality of the agglomerates, see Fig 4 and 5.

[032] Flow measurement according to Hall Flow ISO 4490 for powder dried according to invention gave a value of 31-32 s and for the known technique a value of 34-35 s.

[033] The principles, in preferred embodiments of the present invention have
5 been described in the foregoing specification. However, the invention which is intended to be protected is not to be construed as limited to the particular embodiments disclosed above. Further, the embodiments described herein are to be regarded as illustrative rather than restrictive. Variations and changes may be made by others, and equivalents employed, without departing from the spirit of the
10 present invention. Accordingly, it is expressly intended that all such variations, changes and equivalents which fall within the spirit and scope of the invention be embraced thereby.